“Metal Oxy – Halide Nanostructures: Chemical synthesis, Properties, and Electrochemical Measurements”

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1. Introduction

The transition towards sustainable energy systems has underscored the critical need for efficient energy storage and conversion technologies.Since the world increasingly is turning towards renewable energy sources, such as solar and wind, the inherent intermittency of these resources presents significant challenges for energy supply stability. Energy storage systems play a vital role in bridging the gap between energy generation and consumption, ensuring surplus energy can be stored and used during period of high demand or low generation. Moreover, a rapid expansion of electric vehicles and portable electronic devices have further amplified the demand for advanced energy storage solutions/technologies that offer higher performance, reliability, and longevity. Consequently, the development of innovative materials and technologies for energy storage and conversion becomes paramount to enable a sustainable energy for the future [1]. In this context, researchers are continuously striving to develop advanced systems under the aim of alleviating the growing demands for energy storage devices, such as supercapacitors and batteries [2]. These efforts are primarily focused on improving the efficiency and performance of these devices to meet the escalating energy needs driven by rapid technological advancements. Key areas of exploration include alternative materials and architectures designed to; enhance energy density, extend cycle life, and accelerate charging rates, making energy storage solutions more effective for applications ranging from electric vehicles and renewable energy integration for portable electronics [3]. Additionally, advancements in nanotechnology and materials science are paving the way for designing novel electrode/catalyst materials that exhibit superior electrochemical properties [4]. By leveraging these technologies, scientists are desiring to create energy storage systems that not only meet current demands but also anticipate future energy needs [5]. Furthermore, multi-functional catalysis materials that combine more compact energy storage and efficient conversion capabilities are being investigated [6]. Through these innovative approaches, the goal is to achieve a sustainable energy ecosystem that can effectively support the transition to greener technologies while minimizing reliance on traditional energy sources [7]. Along with the supercapacitor, the splitting of the water through hydrogen evolution and oxygen evolution reactions (HER and OER) is another hopeful approach to simplify the energy request of the world with effective approach. Apart from the contribution to the energy demand, the foremost benefit of the water splitting is zero emission of CO_2 [8]. However, the choice of electrode materials is crucial in achieving high performance in both energy storage and conversion applications [9]. Recent advancements in materials science have led to the exploration of innovative electrode materials, including nanostructured composites and hybrid materials, which exhibit enhanced electrochemical properties. Additionally, the compatibility of the electrode/catalyst materials with the electrolyte and the overall architecture of the device plays a significant role in optimizing performance. By focusing on these critical factors, researchers aim to develop next-generation energy storage and conversion systems that can meet the growing demands of modern technology while promoting sustainability [10]. Therefore, based on the literature survey, a lot of materials have been prepared and used as

electrode/catalyst materials including metal oxide, hydroxide, chalcogenide, etc [11]. These materials show acceptable performance in both supercapacitor and water-splitting application but stile-facing whether cycling stability or showing IR drop [12]. Bismuth, antimony, iron, cobalt etc.,-based electrodes may fill this gap due to their astonishing features including positive/negative potential window, high conductivity, earth abundance etc. [13].

In the past decades, energy storage devices have drawn widespread attention in diverse fields including hybrid electric vehicles and smart portable electronics. By taking advantage of two different electrode materials, asymmetric supercapacitors (ASCs) can extend their operating voltage window and display more excellent energy-storage capacity than those of symmetric supercapacitors. Recent developments have demonstrated that transition metal sulphides (TMSs) have emerged as a promising class of materials for ASCs due to their low-cost, fascinating redox reversibility and electronic conductivity [14].

Electrochemical hydrogen evolution

The development of sustainable clean energy captures great attention of scientists in recent years due to the ever-increasing environmental pollution and energy requirements. Molecular Hydrogen (H_2) is considered as a promising candidate to meet global terawatt energy requirement in the future. Three principle steps typically occur to convert H^+ to H_2 and to realize the hydrogen evolution reaction (HER), ($2H^+ + 2e^- \rightarrow H_2$) process in acidic media that including the adsorption of protons, electrochemical reduction, and desorption of hydrogen gas [4,5]. Therefore, efficient and durable electrocatalysts are usually required to minimize the overpotential and to reduce the free energy barriers (i.e., ΔG_H) of adsorbed hydrogen species, which make the HER process more prone to proceed efficiently in both thermodynamics and kinetics.

In general, chemical reactions occur faster in the presence of a catalyst because the catalyst provides an alternative reaction pathway with lower activation energy than the non-catalyzed mechanism. In catalysed mechanisms, the catalyst usually reacts to form a temporary intermediate, which regenerates the original catalyst in a cyclic process. A substance which provides a mechanism with higher activation energy does not decrease the rate because the reaction can still occur by the non-catalysed route. The added substance which does reduce the reaction rate is not considered a catalyst but a reaction inhibitor. Catalysts may be classified as either homogeneous or heterogeneous. A homogeneous catalyst is one whose molecules are dispersed in the same phase (usually gaseous or liquid) as the reactant's molecules. A heterogeneous catalyst is one whose molecules are not in the same phase as the reactants,

which are typically gases or liquids that are adsorbed in to the surface of the solid catalyst. Enzyme e.g. and other biocatalyst e.g. are often considered as a third category [18].

2. Literature review

Nanostructures of nickel oxy-sulphide ($Ni_xO_yS_{1-y}$) can be created by modifying the surface of three-dimensional nickel-foam with different concentrations of ammonium sulphate. These nanostructures have high power density, energy density, and improved water splitting performance for hydrogen/oxygen evolution in electrochemical supercapacitors and electrochemical catalysis. The reaction mechanism and phase purity of NixOyS1-y are validated by several methods. At 0.02 M concentration, the NixOyS1-y nanostructured electrode exhibits a high specific capacitance (SC) of 5400 F g-1 at 3 A g-1, even after suffering 2000 redox cycles. The NixOyS1-y//NixOyS1-y symmetric supercapacitor device generated a vibrant "CNED" panel with 42 LEDs with maximum luminance, making it useful in home/domestic appliances. Additionally, catalytic activity at 5 mV s-1 result in a current density of 10 mA cm-1 for hydrogen and oxygen evolution reactions, resulting in 217 mVcm-2 and 153 mV cm-2 overpotentials, respectively[19]. Yan et al., synthesized vanadium-doped nickel sulphide nanoflowers of having 2072 F g−1 SC at 1 A g−1 current density with 86.4 % capacitance retention[20]. Al-Abawi et al., prepared a binder-free sphere-shaped interconnected nickel sulphide nanostructure with 88 % cycling stability which adduced an SC of 694.0 F g−1[21]. Bimetallic oxide nanostructures of Gdx; Sb_2O_4 (x = 5, 8, 10 wt. percent) appeared as a novel electrode material for batteries, according to this study, since they have a high specific capacity and cyclic stability. They used a co-precipitation technique for synthesising Gdx (x = 5, 8, and 10% by weight): Sb_2O_4 with excellent electrochemical characteristics. XRD, SEM, UV-Visible, BET, energy dispersive spectroscopy, and XPS spectroscopic examinations were used to evaluate Gd; Sb_2O_4 produced by co precipitation. The specific capacitance of Gd; Sb_2O_4 (10 wt. percent) in 6 M KOH aqueous solution was 958 mA.h/g at a current density of 0.15 A/g, with a capacitance retention rate of 86.5 percent after 1000 cycles. They presented a simple approach for synthesising Gdx; Sb_2O_4 NS, which are promising electrode materials for high-performance super capacitor applications [11]. Jin W et al. studies recent advances of porous transition metal-based nonmaterial for electrochemical energy conversion and storage applications. In these studies, discussed the recent achievements in porous transition metal-based nonmaterial for electrochemical energy storage and conversion, including conventional oxides, sulphide, phosphide, and carbides. They have also, done investigated the current obstacles and future

possibilities of porous transition metal-based nonmaterial in electrochemical energy storage and conversion applications, as well as the designed, manufacturing, and application performance of nonmaterial are illustrated and compared [13]. Sakthivel *et al*. reported synthesis and characterization of bimetallic nickel-cobalt chalcogenides ($NiCoSe_2$, $NiCo_2S_4$, and $NiCo_2O_4$) for non-enzymatic hydrogen peroxide sensor and energy storage: Electrochemical properties dependence on the metal-to-chalcogen composition. In this work, they employed a simple hydrothermal approach to show the synthesis of $NiCoSe_2$, $NiCo_2S_4$, and $NiCo_2O_4$ with various architectures, using electrode materials for electrochemical sensor and super capacitor applications. As findings clearly show Ed superior $NiCoSe_2$ activity of than $NiCo_2O_4$ and $NiCo_2S_4$ in the electrochemical detection of H_2O_2. H_2O_2 was detected with an ultra-low detection limit (0.03 mM) and sensitivity (8.46 mA mM^{-1} cm^{-2}) using the $NiCoSe_2$ modified electrode. Finally, they discovered that $NiCoSe_2$ is an excellent electrode material for electrochemical sensors and supercapacitors [5]. Sharma and Basu 2021 reported Fabrication of centimetre-sized Sb_2S_3/SiO_2 monolithic mimosa pudic nanoflowers for remediation of hazardous pollutants from industrial wastewater. Here, 'mimosa pudic' flower-like Sb_2S_3/SiO_2 monoliths were prepared by impregnation of antimony chloride solution in silica monoliths followed by treatment with potassium thiocyanate, refluxing and calcination. The nanoflower-like morphology of the synthesized monoliths was verified using field emission scanning electron microscope images. The purity/crystallinity and the elemental state of the sulphide were confirmed using X-ray diffraction analysis and X-ray photon spectroscopy whereas energy dispersive spectroscopy verified the homogeneous elemental distribution of the catalyst. The diffuse reflectance spectroscopy and photoluminescence spectroscopy confirmed that Sb_2S_3/SiO_2 monoliths had a narrow band gap (~1.57 eV) and low recombination rate of photoinduced charge carriers. Thermal stability of the materials was also investigated by thermogravimetric analysis [16]. Zhou *et al.* reported acetylacetone-directed controllable synthesis of Bi_2S_3 nanostructures with tuneable morphology. In this report, bi_2S_3 microscopic and solve thermally micro flowers in the acetylase solution were uniformly synthesized by pyrolysis of the Bi_2S_3 dithizone complex without any Molds or surfactants. Bi_2S_3 microscopy was manufactured consisting of nanorods with a diameter of 20-40 nm at 180 °C for 12 h . In similar conditions at 240°C for 3 days, micro flowers consisting of nanowires were obtained with lengths of up to several micrometres and a diameter of 20-40 nm. Here electrochemical experiments used by Bi_2S_3 in a lithium-ion battery indicated that the first discharge capacity of Bi_2S_3 microphones could reach about 148 mA/h [17]. Nagappagari *et al.* studied Enhanced photoelectrochemical activity using $NiCo_2S_4$ / spaced

TiO_2 nanorod heterojunction. In this study, a well-aligned spaced TiO_2 nanorod arrays were uniformly grown on a fluorine-doped tin oxide substrate, and thermal exfoliation of TiO_2 nanorods, which appeared as small islands, was investigated. The calcination time, which ranged from 1 to 5 h at 400 °C, had a significant impact on the agglomerated TiO_2 nanorod islands, which tended to be uniformly exfoliated as spaced nanorods and eventually improved the PEC performance. the heterojunction formation with $NiCo_2S_4$ via the successive ionic layer adsorption and reaction (SILAR) method, substantially improve PEC performance, owing to reduced charge carrier recombination at the interface of the heterojunction. they succeeded in improving the photoelectrochemical performance of TiO_2 by heterojunction construction with $NiCo_2S_4$ nanolayers, as well as narrow optimization of exceptional parameters [19]. Moreover, chalcogenides of non-noble metals such as Ni, Co, Fe Mo, and W are being employed in electrocatalytic processes like hydrogen evolution reaction (HER) and oxygen evolution reaction (OER). Nickel chalcogenides have attracted considerable interests due to a high cost and limited quantity of iridium- and ruthenium-based materials for OER performance[22].

3. Methods

It is important branches of inorganic synthesis. Hydrothermal technique is not just confined to the syntheses and growth of conventional and advanced materials but also take a very broad shape, covering several interdisciplinary branches of science and widely applied.

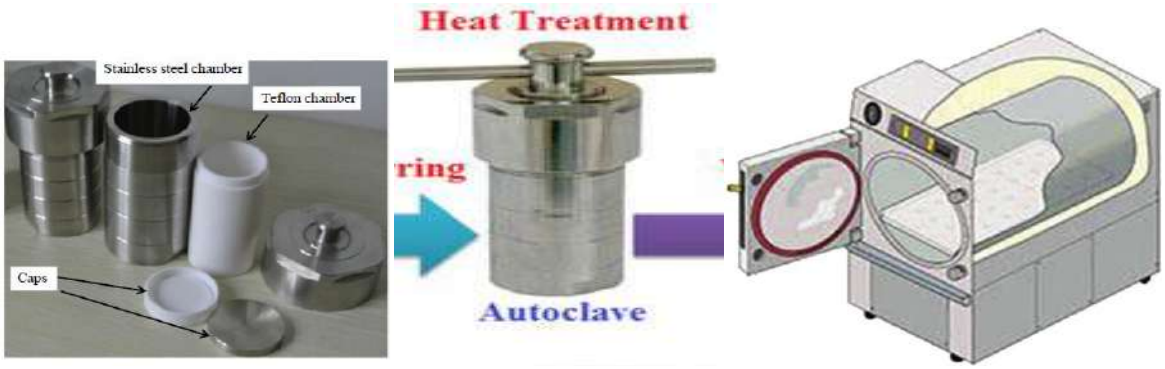

Fig 1: Diagram showing the part of hydrothermal chemical method.

Hydrothermal method for crystallizing substances from high-temperature aqueous solutions at high vapour pressures; also termed “hydrothermal method.’’ The term “hydrothermal” is of geologic origin. Hydrothermal synthesis can be defined as a method of synthesis of single

crystals that depends on the solubility of minerals in hot water under high pressure. The crystal growth is performed in an apparatus consisting of a steel pressure vessel called an autoclave in which a nutrient is supplied along with water. A temperature gradient is maintained between the opposite ends of the growth chamber. At the hotter end the nutrient solute dissolves, while at the cooler end it is deposited on a seed crystal, growing the desired crystal [22].

4. Physical characterizations

Diffraction analysis with X-rays (XRD). The diffraction method is also used to distinguish between crystalline and nanocrystalline materials, and this spectroscopic technique is utilized to identify the crystal's atomic and molecular structure. This spectroscopic approach is also used to distinguish the nature of crystalline materials from nanocrystalline materials, and it is used to detect the atomic and molecular structure of crystals. Scanning electron miscroscopy (SEM) is a characterization technique that uses a focused electron beam to scan a solid sample from the optical to nanoscale range. In these types of characterization processes, secondary electrons are released from the sample and collected to create a secondary emission area map. Field-emission microscopes will be used to determine the external morphology (texture), chemical composition, and crystalline structure of materials. Raman spectroscopy uses laser light to investigate molecular structure. For every million photons distributed, a single photon will interact with the vibrational states of a sample molecule and emit light of a distinct wavelength. In addition to IR spectroscopy, Raman systems can be used to explore molecular structure.

In transmission electron mocroscopy, the electrons are focused directly on the substance. The HR examines the material as a whole, rather than just the surface. It has a magnification range of 50 million and displays a two-dimensional image. An electron beam is fed through an ultrathin material, interacts with it as it passes, and forms an image in a HR with a resolution of 0.5 Angstrom. In both physical and biological sciences, TEM characterizations are used. The high-resolution electron microscope will be used to determine the particle size of nanoparticles. They work in a variety of academic, scientific, and industrial sectors as well. X-ray photoelectron spectroscopy (XPS) is a surface-sensitive quantitative spectroscopic method that determines the elemental composition, empirical formula, chemical state, and electronic state of the elements present in a material in parts per thousand. Simply said, XPS is a valuable

measuring technique since it displays not only what components are included inside a film, but also what other elements they are bound to.

5. **Electrochemical measurements**

A three-electrode system on an electrochemical workstation will be used to assess both water spitting and supercapacitor performance. The entire measurement procedure will be carried out in an electrolyte (e.g., 0.5 mL H_2SO_4) with a counter electrode, an Ag/AgCl (in saturated KCl solution) reference electrode, and a catalyst-modified GCE that was generated using the methods above as the working electrode. CV is an electrochemical method for determining the current that occurs in an electrochemical cell when the voltage exceeds that predicted by the Nernst equation. CV may be used to investigate qualitative information about electrochemical processes under a variety of situations, such as the existence of intermediates in oxidation-reduction reactions and a reaction's reversibility. CV may will be used to estimate a system's electron stoichiometry, an analyst's diffusion coefficient, and the formal reduction potential, which can be used as a tool for identification. Electrochemical impedance spectroscopy (ESI) is an electrochemical approach for determining the impedance of a system in relation to the frequency of the AC potential. It's also a useful instrument for studying chemical and physical processes in both liquids and solids. EIS is a potent non-destructive tool for researching microbial fuel cells that can complement current approaches. Galvanostatic charge-discharge (GCD) is a key metric in supercapacitors that provides all information about the electrode material, such as specific capacitance, stability, power and energy densities, and so on. In a charge-discharge curve with a fixed potential window, the current density remains constant, but it may be modified to get varied discharge time values between the two potentials. The curve in the charge process can start at zero time or any value within the potential window's range and climb linearly to the desired potential. Then it reveres its direction to return to the original value within a certain time frame. Electrochemical hydrogen evolution reaction presents polarisation curves obtained using a variety of sweeping potentials. The j–t curve of chronoamperometry is measured at a constant voltage. In the same scenario, electrochemical impedance spectroscopy is acquired at a certain over potential, frequency range, and amplitude. Oxygen evolution reaction is a limiting reaction in the process of producing molecular oxygen by chemical reactions, such as water oxidation during oxygenic photosynthesis and water electrolysis into oxygen and hydrogen. The success of a variety of renewable energy technologies, including solar fuel generation and metal–air batteries, hinges

on the development of better catalysts for the OER. Oxygen is released from water by oxygenic photosynthesis, water electrolysis, and the thermal decomposition of different oxides.

6. Objectives and topic of research

Therefore, during Ph.D. course at the school of Chemical Sciences, Swami Ramanand Teerth Marathwada University, Nanded, India, it is an objective of work to synthesis metal oxy-halide nanostructures using cost effective chemical method.

- ✓ These nanostructures will be excplored for their physical and chemical properties by various means
- ✓ These nanostructures will be envisahed in electrochemical studies for knowing their usefulness in either electrochemical supercapacitors or electrocatalysis.
- ✓ Efforts will be made to understand the performance change from oxide to halide as oxy-halide both theoretically and experimentally.

Based on in-depth review, manuscripts of world class standard will be prepared and published in reputed journals.

The wide applications of nanostructured are based on the progress in the synthesis of new nanostructured materials of different sizes and new morphologies. The best materials that can be used as an electrode in store energy, as well as supercapacitor, are Heterojunction materials due to their wide potential window, highly redox reactions, distinct oxidation states, high conductivity, remarkably low over potential, good thermal stability, long cycle life, and high-rate capability as well as it is an environment friend. Therefore, further developing new materials with low overpotential and better performance relative to existing electrode materials is the most important technique to overcome these challenges. And also for the following metal oxides/metal sulphides will be study as:

1) Synthesis of various metal oxy – halide nanostructures electrode/catalyst materials
2) Their reaction mechanims will be proposed based on FTIR and XRD analyses.
3) Measurement of their physical characterization like structure, composition, surface appearance, optical studies etc.
4) Measurement of their electrochemical characteristics like CV, CCB, life time, impedance spectroscopy, measuring specific capacitance.
5) Measurement of water splitting properties such as OER, HER for measuring overpotential, Tafel slope and stability etc.

6) Writing manuscripts, and thesis for publishing in international reputed journals and for the award of Ph.D. in Chemistry.

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